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Faculdade de Odontologia de Piracicaba

LUCAS COSTA DE MEDEIROS DANTAS

“EFEITO DE MÉTODOS DE AVALIAÇÃO NA RESISTÊNCIA DE UNIÃO ENTRE CERÂMICA ODONTOLÓGICA E CIMENTOS RESINOSOS”

“*EFFECT OF MECHANICAL METHODS OF EVALUATION ON THE BOND STRENGTH BETWEEN DENTAL CERAMIC AND RESIN CEMENTS*”

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UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA

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"*EFFECT OF MECHANICAL METHODS OF EVALUATION ON THE BOND STRENGTH BETWEEN DENTAL CERAMIC AND RESIN CEMENTS*"

Tese apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos para obtenção do título de Doutor em Materiais Dentários.

Thesis presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for degree of Doctor in Dental Materials.

Orientador: Prof. Dr. Simonides Consani

Coorientador: Prof. Dr. Lourenço Correr Sobrinho

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A Comissão Julgadora dos trabalhos de Defesa de Tese de Doutorado, em sessão pública realizada em 06 de Julho de 2015, considerou o candidato LUCAS COSTA DE MEDEIROS DANTAS aprovado.

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RESUMO

Diferentes métodos de ensaio para verificação da resistência de união entre materiais odontológicos são descritos na literatura como meios de predizer o comportamento destes materiais em meio oral. Entre os métodos utilizados, os mais mencionados são os de microtração e microcisalhamento. Apesar de amplamente estudados, existe uma grande variação nos protocolos de confecção de espécimes e de ensaio mecânico, dificultando a comparação entre os estudos. O objetivo neste estudo foi avaliar o comportamento da interface de união entre cerâmica odontológica e dois cimentos resinosos sob influência de diferentes ensaios de resistência de união, potências de fotoativação e realização de ciclagem térmica. Utilizando o método de elementos finitos, no capítulo 1, dois modelos, um simulando o ensaio de microcisalhamento e o outro o ensaio de microtração, foram analisados quanto à distribuição de tensões na região de união dos espécimes. Assim, no capítulo 2, o ensaio de microcisalhamento foi utilizado para avaliar a resistência de união entre dois cimentos resinosos e uma cerâmica odontológica variando a potência do aparelho fotoativador, e a simulação de envelhecimento por meio de ciclagem térmica. Por meio de teste *in vitro,* os ensaios de microtração e microcisalhamento foram utilizados para avaliar a resistência de união entre os mesmos materiais e com os mesmos protocolos de confecção, variando o tipo de cimento resinoso e a potência do foto ativador. O método de elementos finitos revelou a presença de uma tensão uniaxial para o modelo de microtração. Nos ensaios laboratoriais o ensaio de microcisalhamento resultou em maiores valores de resistência de união. O método de envelhecimento de amostras diminuiu a resistência de união. Desta forma, conclui-se que a utilização da baixa potência de fotoativação favoreceu o resultado de resistência de união imediato e que o ensaio de microtração performado neste estudo apresentou tensões melhores distribuídas indicando melhor utilização no ensaio.

Palavras-chave: Cimento resinoso, Cerâmica odontológica, Resistência de união, Microtração, Microcisalhamento, Ciclagem térmica.

ABSTRACT

Different test methods to verify the bond strength of dental materials are described in the literature as a means to predict the behaviour of these materials in the oral environment. Among the methods used, the most mentioned are the microtensile and microshear. Although widely studied, there is a large variation in the preparation of specimens and mechanical testing protocols, making it difficult to compare studies. The aim of this study was to evaluate the behaviour of the adhesive interface with two resin cements and curing powers and performing thermal cycling, assessed by two different tests of bond strength. Using the finite element method, in chapter 1, two models were made, one simulating the test microshear and the other microtensile test were analysed for the stress distribution in the adhesive region of the specimens. Thus, microshear test, in chapter 2, was used to evaluate the bond strength of two resin cements and dental ceramics by varying the power of curing unit device and the simulation of ageing by thermal cycling. By the means of *in vitro* assay tests microtensile and microshear were used to evaluate the bond strength of the same materials and with the same protocols of specimens preparation by varying the type of resin cement and the power of the photoactivator. The finite element analysis revealed the microtensile model a uniaxial stress. In laboratory, microshear results in higher values of bond strength. The ageing method promoted reducing in bond strength values, while the low power of photoactivation increases these values. It may be concluded, that low intensity of photoactivation leads to an immediately higher bond strength and

microtensile bond strength test performed in this study showed the stresses distributed in correct axis indicating better use to bond strength evaluation.

Key-words: Resin cement, Dental ceramic, Bond strength, Microtensile, Microshear, Thermocycling.

SUMÁRIO

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“A mente que se abre a uma nova ideia jamais voltará ao seu tamanho original”

Albert Einstein

INTRODUÇÃO

As restaurações indiretas em cerâmicas apresentam vantagens em relação às restaurações diretas em resina composta, como maior resistência ao desgaste, menor susceptibilidade à pigmentação e melhor capacidade de mimetizar o esmalte dental (1). A união entre as restaurações cerâmicas e a estrutura dental pode ser obtida pela associação de tratamentos da superfície da cerâmica e o uso de cimentos resinosos (2-4).

Para que se alcance o sucesso das restaurações cerâmicas depende em grande parte do tipo de cimento resinoso utilizado para garantir união efetiva entre material restaurador e estrutura dentária, proporcionando boa adaptação marginal (5). Este tipo de material restaurador permite união ao substrato dental por meio da fixação adesiva associada ao silano, sistema adesivo e cimento resinoso (6). Os cimentos resinosos podem ser classificados de acordo como tipo de condicionamento: total ou auto-adesivo. Cimentos resinosos que empregam o condicionamento total necessitam do uso de sistemas adesivos para hibridização do substrato dental antes do emprego do cimento resinoso. Os cimentos resinosos auto-adesivos são capazes de se unir aos tecidos dentais sem a necessidade prévia do uso de sistemas adesivos (7, 8).

De outra forma, os cimentos resinosos podem ser classificados de acordo com o modo de ativação: fotoativado, quimicamente ativado e de dupla ativação. Cimentos resinosos ativados somente pela exposição à luz oferecem vantagens, como maior tempo de trabalho e melhor estabilidade de cor. Os cimentos resinosos quimicamente ativados apresentam a vantagem de serem utilizados para a cimentação em locais em que a luz ativadora da polimerização tem acesso restrito (6, 9). Assim, quando a fotoativação do cimento resinoso for realizada de forma indireta, alguns aspectos devem ser levados em consideração, como: a medida que a espessura do material restaurador indireto aumenta, a absorção de luz diminui e a sua dispersão aumenta, reduzindo a quantidade de energia fornecida ao cimento resinoso pelo fotoativador (10-15). De acordo com Kurachi (2001), existe um efeito atenuador da luz proporcional à espessura da cerâmica e da opacidade do material restaurador indireto, reduzindo as propriedades mecânicas dos cimentos resinosos, o que pode comprometer a união entre o cimento resinoso e o material restaurador (16).

Desse modo, torna-se importante otimizar os métodos de fotoativação dos cimentos resinosos com a finalidade de melhorar o desempenho clínico dos materiais, pois maior conversão dos monômeros é imprescindível para melhorar o desempenho desses materiais (9), já que a polimerização inadequada do cimento resinoso pode estar associada à propriedade mecânica inferior, alta absorção de água e solubilidade, além da instabilidade de cor (6).

Em relação à avaliação da resistência de união entre materiais restauradores e diferentes substratos, os testes convencionais de tração e cisalhamento têm sido utilizados há alguns anos, embora tenha sido demonstrado que os resultados obtidos por essas metodologias não representam com fidelidade a resistência de união para os materiais testados (17, 18). Isso ocorre pelo fato de os espécimes utilizados nesses testes serem mais propensos a ter defeitos incorporados à interface adesiva ou aos substratos devido às grandes dimensões dos mesmos (19). Além disso, pelo modo como os espécimes são carregados e pela própria geometria destes, tensões não uniformes podem ser induzidas nas regiões de interesse, levando a grandes variações nos resultados obtidos (20-23).

Na tentativa de superar as limitações destes testes, foi proposta a avaliação da resistência de união interfacial em áreas adesivas reduzidas (1,0 mm^2) utilizando o teste de microtração (24). Com esta modificação foi possível a obtenção de vários espécimes a partir de um único dente, além da possibilidade de mensuração da resistência adesiva em diferentes regiões do substrato dental. As vantagens desta metodologia na avaliação da resistência de união de diferentes materiais foram ressaltadas por inúmeros autores (20, 25-27) e atualmente, este é o teste laboratorial mais comumente utilizado para este fim. Entretanto, a popularização desta metodologia permitiu que inúmeras modificações fossem introduzidas (25).

Assim como ocorreu com o teste de microtração (20, 27), a constante utilização do ensaio de microcisalhamento (28) levou à proposição de modificações na abordagem inicial sugerida para esta metodologia (29-31). Desta forma, materiais similares avaliados em diferentes configurações deste teste podem apresentar resultados conflitantes entre si. Alguns parâmetros importantes do teste de microcisalhamento são a forma de posicionamento dos espécimes e a direção do carregamento aplicado sobre os mesmos durante o ensaio (32). Da mesma forma, as pontas de carregamento também podem influenciar os resultados de resistência de união obtidos utilizando o microcisalhamento (33).

Independente do teste empregado para verificação da resistência de união existe grande variação nos dados obtidos laboratorialmente (34, 35). Esse fato pode ser mais bem compreendido por meio de análises utilizando o método de elementos finitos, nas quais se observa o acúmulo de tensões nas regiões de interesse em diferentes testes devido a variáveis envolvendo geometria, modo de carregamento, propriedade dos materiais e forma de preparo dos espécimes (20, 22, 23, 27, 34, 36-42).

A união dos materiais cerâmicos aos cimentos resinosos tem sido amplamente estudada e diferentes métodos para avaliar a resistência de (microtração e microcisalhamento) tem sido empregados (2, 4, 8). Por isso, avaliação da viabilidade e comparação dos resultados entre os métodos de ensaio devem ser realizados. Entretanto, dúvidas permanecem a respeito do efeito da fotoativação indireta sobre as propriedades mecânicas dos cimentos resinosos fotoativados através de diferentes espessuras da cerâmica.

Os objetivos do presente estudo *in vitro*, composto por dois artigos científicos, foram:

1. Verificar o efeito da termociclagem e da potência do fotoativador na resistência de união de dois cimentos resinosos à cerâmica odontológica por meio do ensaio de resistência de união (Capítulo 1);
2. Analisar a resistência de união obtida por diferentes tipos de ensaio mecânico (Capítulo 2).

CAPÍTULO 1 – Bond strength of dental ceramic to resin cements: laboratorial and finite element analyses.

ABSTRACT

Objectives: The aim of this study was to evaluate the effect of photo-activation power modes on resin cements in relation to microtensile and microshear bond strength tests and the stress distribution by finite element analysis. *Methods:* Forty plates of di-silicate based ceramic (8.0 x 8.0 x 2.0 mm in height) were confectioned and randomly divided according to resin cement, and divided again according to the photo-activator power used to prepare the specimens. Following, they were divided according to the microtensile and microshear tests. Microshear specimens were made with Tygon tube matrices. The resin cements were inserted into the tube and after 5 minutes photo-activated for 40 seconds. The specimens were stored in 100% relative humidity and 37ºC for 24 hours. After storage, the tubes were carefully removed. For microtensile (MtBT) specimens, twenty blocks of composite resin were made and cemented on the ceramic plates with the resin cement 500gf was applied on the blocks for 5 minutes and photo-activated by 40 seconds. The specimens were stored in 100% relative humidity at 37ºC for 24 hours. After the storage, the blocks were trimmed in beams of 1.0 mm^2 section. The bond strength tests were performed in a mechanical test machine at 0.5 mm/min cross-head speed. Data (MPa) were checked for homoscedasticity and analysed using one-way analysis of variance (ANOVA) and Tukey's test ($p<0.05$).

Two 3D models were generated and meshed using eight-node hexahedral elements. All structures and materials were considered homogeneous, linear-elastic and isotropic. For microshear bond test (μSBS), the loading point was generated according to the laboratory test simulating an orthodontic-looped wire. *Results:* Three-way ANOVA showed significant differences for testing time ($p<.001$), resin cement ($p=.034$) and interaction between photoactivation power and testing time ($p<.001$). Although the lower values were obtained to the higher power mode, stresses analysis have shown different behaviours for bonding interface area. For maximum principal value of stress (MP), von Mises stress (VM) and Y axis of stress the tensile and compression stresses were highlighted. *Conclusion:* Microshear bond strength revealed higher values than the microtensile test for lithium disilicate dental ceramic. Perpendicular stresses to the load direction were found mainly for μSBS model, where tensile strength was revealed in the area near to the load application.

Key-words: Dental ceramics, Resin cement, Photoactivation, Microshear, Microtensile, Testing parameters, Bond strength

INTRODUCTION

The knowledge on the properties of dental materials is very important to characterize the behaviour under different test conditions and at the oral environment. Laboratorial methods used to assess the mechanical properties of these materials have been established in some dental studies [1,2]. Mechanical tests are routinely applied to characterize the behaviour of dental materials or to evaluate the mechanical properties. Assays verifying bonding quality and strength of adhesive systems-resin cements to dental substrates-restorative materials are the tests most commonly employed for dental materials [3].

Conventional tensile and shearing tests have been applied for several years in dental materials evaluation, although it has been shown that the results obtained with these methodologies do not completely represent the bonding strength of the testing materials with great accuracy [4,5]. This occurs because the specimens used in the tests are more prone to have failures incorporated at the adhesive interface or at the bonding substrates due to their increased dimensions [6]. Moreover, owing to the loading mode and the geometry of the specimens used on these methodologies, uneven stress can be induced at the bonding interfaces, promoting large variations in the results [3,7-9].

In an attempt to overcome the limitations of the previous bonding tests, previous study evaluated the interfacial bond strength from smaller bond test areas (1.0 mm^2) using the microtensile approach [10]. With this methodology, it became possible to obtain several specimens from a single tooth, measuring bond strength in different regions while reducing scatter and achieving adhesive failures in the majority of specimens. The advantages of this approach for testing bonding interfaces have been highlighted by numerous studies and today it is the most used mechanical test for this purpose [3,11-13]. However, the dissemination of this methodology allowed adaptations to be suggested through the original approach [11], resulting in conflicting results.

As occurred with the tensile/microtensile methods, the tendency toward evaluating bonding interfaces in reduced adhesive regions in order to incorporate less failures and variables during the test was also conceived for the shearing approach. The shearing test using specimens with adhesive interfaces inferior to 1.0 mm^2 was initially proposed, being nominated “microshearing” [14]. Another microshear modality was also suggested for checking the bond strengths of dental materials, using very small cylinders of resin-based cements (ø 0.75 x 0.5 mm) bonded to different substrates (dentin, enamel, ceramics, etc.) [15-17]. With this methodology, it became possible to prepare multiple bonding specimens in a single ceramic surface or even at a single tooth region, without requiring additional procedures that may cause test variables, such as the trimming of the adhesive region necessary in many microtensile specimens [18,19].

Since microshear specimens have reduced dimensions, this test design allows reduced failure incorporation, providing more accurate results for bonding strength evaluation [20]. Some comparative investigations have shown that the microshear test can provide bond strength results as reliable [20], or even more accurate [21,22], than the obtained using the microtensile approach. The viability of this methodology is also demonstrated by its crescent utilization in the recent literature [23-27]. However, independently of the mechanical test used for evaluating bond strengths, large variation exists in the results [28,29]. The popularization of the microshear methodology also allowed modifications to be introduced [16,17,30], what can promote conflicting results between studies.

Therefore, the aim of this study is to evaluate the effect of photoactivation power modes and resin cements in microtensile and microshear bond strength tests and the stress distribution by finite element analysis. The hypotheses are that (1) decreased power mode would result in lower bond strength, (2) different resin cement would result in different bond strength values, (3) no stress would be generated in perpendicular axes to the load, and (4) the stress generated at the interface area would be similar to both tests.

MATERIALS AND METHODS

Two dual-cure resin cements, a self-adhesive cement (RelyX U200, 3M ESPE, St. Paul, MN, EUA) and a conventional cement (RelyX ARC, 3M ESPE) were used. Forty plates of di-silicate based ceramic (IPS e.max Press, Ivoclar-Vivadent, Schaan, Liechtestein) with 8.0 x 8.0 x 2.0 mm in height were confectioned and randomly divided according to the cement (n=20). Then they were divided again according to the photo-activator power (BluePhase G2, Ivoclar-Vivadent) used to prepare the specimens (n=10). Following, were divided according to the microtensile and microshear tests.

Prepare of ceramic surfaces

The ceramic surface for the adhesive procedure were conditioned with 10% hydrofluoric (Dentisply) acid for 20 seconds, rinsed for 60 seconds and dried for 30 seconds following manufacturer's recommendations. Following, the silane agent was applied twice on the ceramic surface, followed by a thin layer of adhesive after one minute from the silane application.

Microshear test

Tygon tubes (0.75 mm inner diameter and 0.5 mm height) were positioned on the plates prepared to adhesive procedures according the distances recommended by previous study [31]. The resin cement was inserted into the tube and photo-activated by 40 seconds 5 minutes after the procedure according to manufacturer's recommendations. The specimens were stored at 100% relative humidity and 37ºC for 24 hours. After the storage procedure, the tubes were carefully removed. The specimens were fixed with glue (SuperBonder, Loctite, Itapeví, SP) in the test device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, EUA) before being tested in a mechanical test machine (EZ-Test, Shimadzu, Tokyo, JAP) at 0.5 mm/min cross-head speed with an orthodontic-looped wire.

Microtensile test

Twenty blocks of composite resin were made and cemented on the ceramic plates with the resin cement and photo-activated for 40 seconds after 5 minutes from the procedure with load of 500 gf according to manufacturer's recommendations. The specimens were stored at 100% relative humidity and 37ºC for 24 hours. After the storage, the blocks were trimmed in beams of 1.0 mm^2 section. Beans were fixed in test device with cyanoacrylate-based resin (SuperBonder) and tested in a mechanical test machine at 0.5 mm/min cross-head speed.

Data were analyzed for homoscedasticity using Shapiro-Wilk test. Three-way analysis of variance (ANOVA) was applied to establish the significance of differences between testing groups followed by Tukey HSD test. All tests were performed at 95% confidence level using a statistical software package (Sygma Plot 12, Systat Software Inc., San Jose, CA, USA).

Finite Element Analysis (FEA)

Two three-dimensional (3D) models were generated and meshed using eight-node hexahedral elements (MSC Mentat 2010, MSC Software Corporation, Santa Ana, CA, USA). All structures and materials were considered homogeneous, linear-elastic and isotropic. The mechanical properties were defined by literature search (Table 1). For the microshear bond test (μSBS), the loading point was generated according to the laboratory test simulating an orthodontic-looped wire.

Table 1 - Mechanical properties of tooth structures and materials used in the FEA: Elastic modulus (*E*) and Poisson's ratio (*v*)

Material	*E* (GPa)	*v*
Lithium disilicate ceramic [32]	120.0	0.25
Composite resin [33]	16.6	0.24
Resin cement [34]	5.1	0.27
Cyanoacrylate glue [35]	6.0	0.3

A 10 N non-linear load was applied to the models. For μSBS the charge was applied in the loading point, and for the microtensile bond test (MtBT) the load was applied in the nodes simulating the specimen positioning in test machine. Static structural analysis was performed considering non-linear contacts and constraints at X, Y and Z axes (Figure 1 b-d-f). For the microtensile test the beam was simulated glued in the test device with constraints in X and Z axes and nodal load in Y axis (Figure 1 a-c-e). Furthermore, plots were made to analyze the stresses for the adhesion area with stress values of centerline of nodes in the interest area. For the μSBS, one line at inner ceramic, other at inner resin cement and other at the interface. For the MtBT, three lines at inner resin cement, since the model presents two interfaces. The results were analysed using von Mises, maximum principal and isolated direction of stresses.

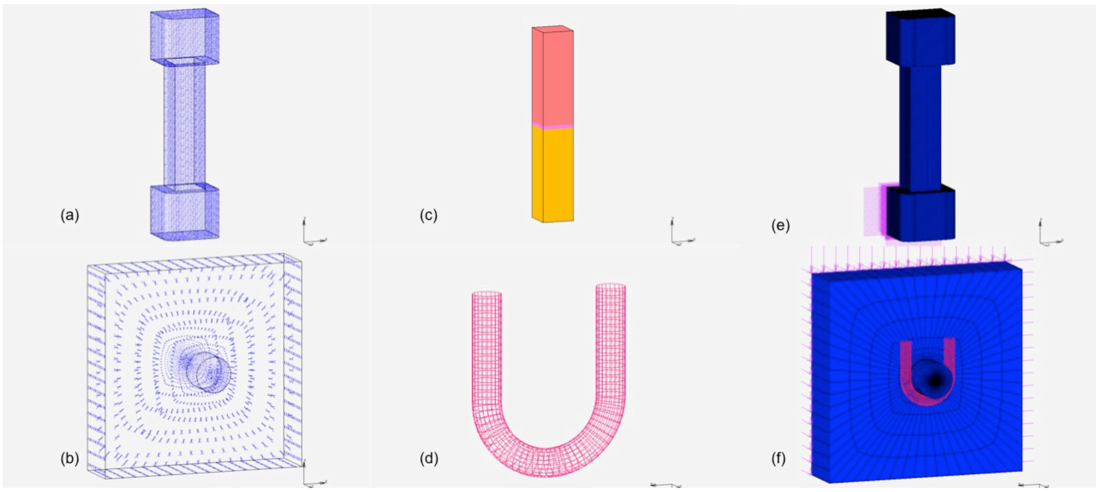

Figure 1 – Finite element models: Mesh generated (a, b); beam to μTBS without cyanoacrylate resin (c); Point load for μSBS (d); Boundary conditions to tests (e, f).

RESULTS

The three-way ANOVA showed significant differences for resin cement ($p<.001$), bond strength test ($p=.037$) and interaction between photo-activation power mode and resin cement ($p<.001$).

Table 2 – Bond strength (MPa) and standard deviation for RelyX ARC and RelyX U200 in relation to power mode for microshear bond strength (μSBS).

Bond strength test	Power mode	Resin cement	
		RelyX ARC	RelyX U200
Microshear	Low	40.84 ± 5.97 Aa	42.01 ± 4.04 Ba
	High	33.38 ± 4.78 Bb	35.60 ± 4.11 Aa

* Different capital letters indicate significant difference between columns and different small letters indicate significant difference between rows (p<.05).

Table 2 shows lower values for the higher power mode to microshear bond strength and Table 3 shows lower values for the lower power mode to microtensile bond strength. The results obtained by different bond strength test showed significant difference (μSBS; 37.96±5.01 and μTBS; 27.96±4.04; p=.037)

Table 3 – Bond strength values (MPa) and standard deviation for for RelyX ARC and RelyX U200 in relation to power mode for microtensile bond strength (μTBS).

Bond strength test	Power mode	Resin cement	
		RelyX ARC	RelyX U200
Microtensile	Low	22.56 ± 6.07 Bb	25.80 ± 6.62 Ab

	High	29.67 ± 4.63 Ba	33.66 ± 1.91 Aa

* Different capital letters indicate significant difference between columns and different small letters indicate significant difference between rows ($p<.05$).

Finite element analyses showed to μSBS higher stresses area next to load application, in opposite region compression stress was found, and shear stress distributed on the interface. To μTBS model, perpendicular stress was almost null and tensile stress distributed on the interface with higher values next to the face of load application (Figure 2).

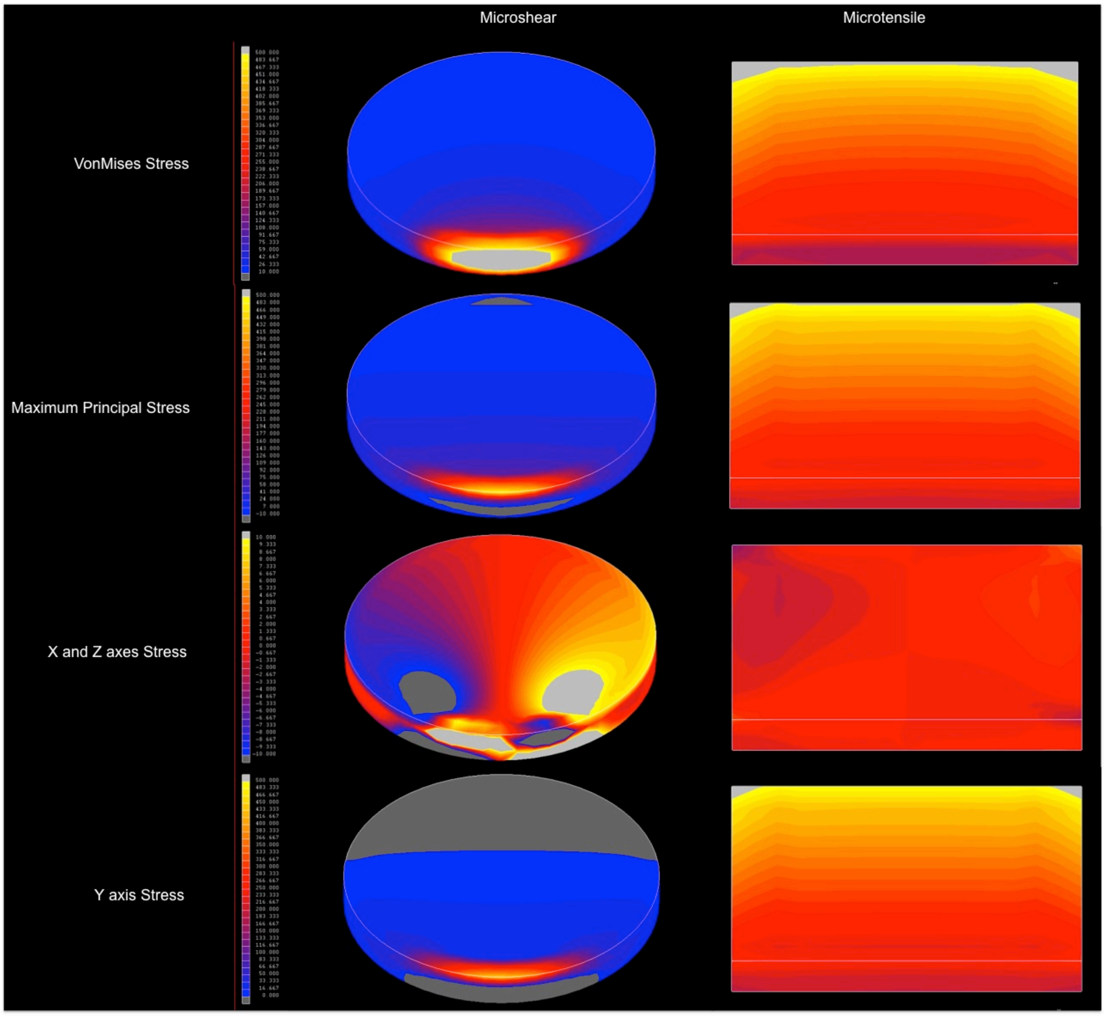

Figure 2 – Finite element results in vonMises stress, maximum principal stress, X and Z axes stress, and Y axis stress analyses according to the μSBS and μTBS designs in the interface between dental ceramic and resin cement.

According to the plots obtained from the nodes is possible to confirm the data showed in images (Figure 2) where for μTBS occurred tensile stress in whole interface area and the shear stress was almost null (Figure 3 – top plot). The tensile stress generated by the loading point at µSBS is revealed in the plot (Figure 3 – bottom plot).

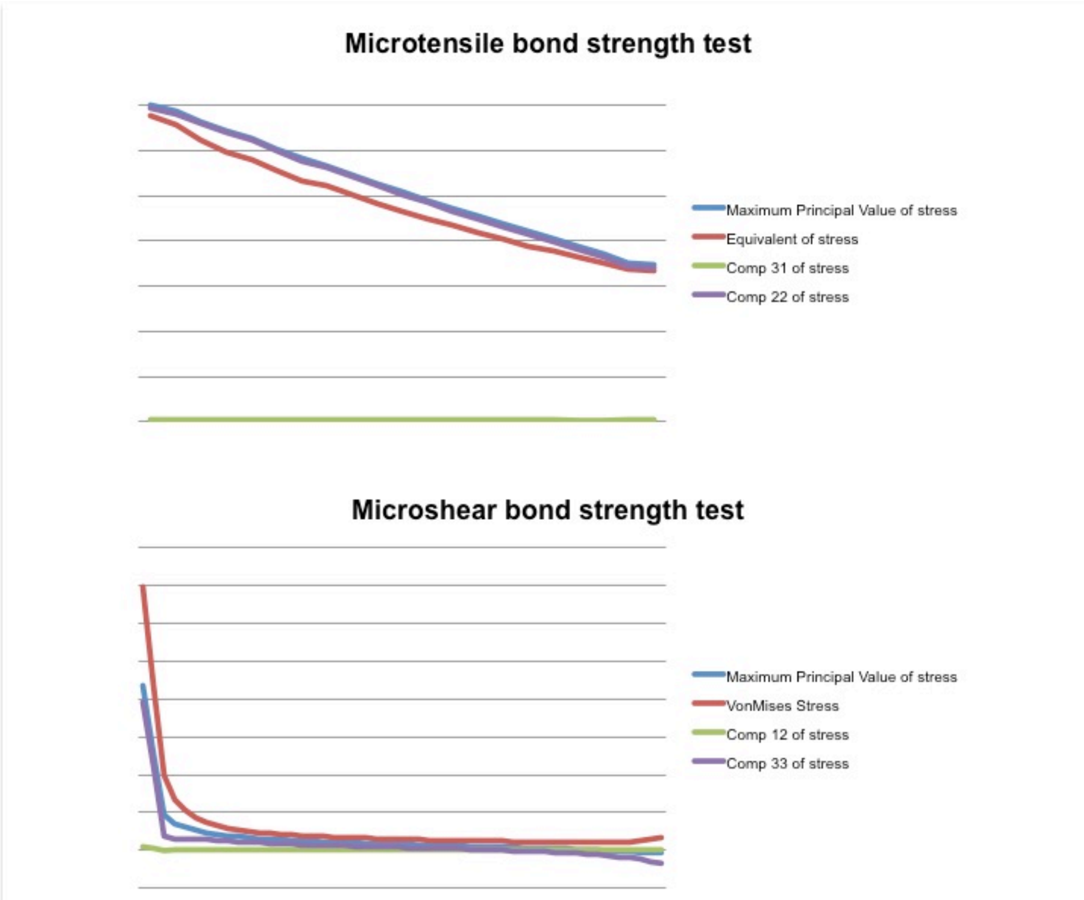

Figure 3 – Finite element results according to the µSBS and µTBS designs in plots of interface area.

DISCUSSION

The hypothesis that decrease power mode would result in lower bond strength values was rejected, since lower values were founded increasing power mode. The hypothesis that different resin cements would result in different bond strength values was accepted. The hypotheses proposed to the FEA were rejected by the investigation, since even near zero values for the stresses perpendicular to the applied load were not zero, particularly to the μSBS where the graph plot shows the stress on the interface near to the loading is very high. This result is indicating that this stress can promote the specimen failure in laboratorial test and likewise. The behaviour of two models were different at the interface even virtue of its mechanical behaviour, the μTBS revealed tensile stress in whole interface since the load promote tensile in the specimen; however, although the load μSBS induces shear stress to the interface were also found tensile stress in many regions.

Microshear testing is a very useful methodology, which allows reduced failures to be incorporated to the specimens, assuring effective bonding assessments [15-17]. Its feasibility has been proved in previous investigations [20,22]. However, large variation still exists in the available bonding results due to lack of standardization of the testing parameters used on bonding studies [3,13]. As seen, small variations in the microshear test parameters can result in different stresses at the adhesive interfaces of the specimens and consequently in distinct shear bond strength values, making it difficult to compare the clinical performance of materials. According to previous study [36], the loading systems produced very distinct stress patterns at the adhesive interface of the microshear specimens, when the orthodontic-looped wire is used tensile stress is promoted in the area near to the initial load applied. Thus, this stress can overlap the desired stresses in the test and the failure occur by tensile and not by shear stress.

The realization of a widely accepted, validated, standardized test method for bond strength testing is an elusive and controversial endeavor [22-24]. Although a consensus or standard approach does not currently exist in literature to elect the test that should be used to each material or condition. The reduced adhesive area in bond strength test shows many advantages, such as smaller test specimens are ‘stronger’ than larger ones due to the lower probability of having a critical sized defect present and aligned in a crack opening orientation relative to the applied load, and this approach have been highlighted by numerous studies and today it is the most used mechanical test for this measures [3,11-13]. However, the dissemination of this methodology allowed adaptations to be suggested through the original approach [11], resulting in conflicting results.

Findings based upon FEA and failure mode analysis of μTBS testing are reported; however, hold true for μSBS and corroborate with the results of this study. These FEA findings were: (1) tensile stresses produced by the bending moment at load application were responsible for fracture initiation, (2) highly non-uniform stress distribution concentrated in the substrate, and (3) a nominally measured bond strength that severely underestimates the true stress the specimen resisted at fracture [30,36,37].

However, it was concluded that microshear bond strength (μSBS) may actually worse represent shear bond strength than the conventional macroSBS test [38]. Increased stress concentration and tensile forces during shear load application were shown when factoring in the relatively thicker adhesive layer, farther load application from the adhesive bond, and the use of lower modulus flowable resin cements (to avoid the introduction of flaws in the small molds required) common to μSBS. In contrast, studies using three-dimensional FEA demonstrated that the tensile forces during loading could be minimized by optimizing specimen dimensions and load application location [15,39,40].

A considerable number of studies presenting microtensile bond strength tests were published in the last decade [11-21]. These authors have described numerous advantages of this ‘microtensile’ methodology to assess bond strength of different materials [6,7]. However, variable methods and parameters (gripping devices, specimen preparation and geometry, and test speed) have been employed by the different laboratories all over the world resulting in bond strength data that can hardly be compared across studies [41,42].

The microtensile bond strength test (μTBS) is the most widely used when the substrate tested are dentin and direct restorations materials, but the difficult to trim a dental reinforced ceramic lead the researches to use a non-destructive assay, as the μSBS. In the current study, the numeric values for μSBS were higher than to the obtained for μTBS with the same material tested. This may occur by the increased stress concentration and tensile forces during shear load application that cause difficult to lead the specimens to adhesive failure.

An important aspect in this investigation occurred when the low power of investigation was performed, since for µTBS the values were significantly different from that obtained for µSBS. This fact probably occurred by the way of specimen confection. For the microshear, the photoactivation was performed directly in the resin cement, whereas for microtensile there was the barrier of the composite block. This obstacle can have drastically reduced the transmitted light intensity, consequently, reducing the resin cement polymerization. This can explain the similarity between the michoshear groups and the difference between the microtensile groups.

The different resin cements used in this investigation represent differences in results. However, a possible chemical incompatibility between adhesive systems with low pH and chemically and dual-polymerizing resin materials are reported.[43] It is known that acidic resin monomers retard the polymerization of chemically/dual-cured composites that are initiated via peroxide-amine type binary redox catalysts.[43] It is possible to infer that in this study the alleged inhibition could affect the results.

CONCLUSIONS

Within the limitations of the present in vitro study, the conclusion can de drawn:

(1) The microshear bond strength revealed higher values than the microtesile when the lithium disilicate ceramic was bonded to resin cement;

(2) RelyX ARC and RelyX U200 materials were similar when the same bond strength test was used;

(3) The photo-activation power showed opposite behavior for the different bond strength tests;

(4) Perpendicular stresses to the load direction were found mainly to the μSBS, where tensile strength was revealed in the area near to the load application;

(5) μTBS was more reliable since the stress generated in the interface is almost uniaxial, whereas μSBS revealed biaxial stresses.

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CAPÍTULO 2 - Effect of thermocycling and photactivation power on the bond strength of different resin cements to a lithium disilicate ceramic.

ABSTRACT

Objectives: This study aimed to evaluate the effects of thermocycling and photoactivation power on the bond strength of two resin cements, conventional and autoadhesive, to a lithium disilicate-based glass ceramic. *Methods:* Forty ceramic plates (8.0 x 8.0 x 2.0 mm) were produced and randomly divided according to the resin cement (conventional or self-adhesive), the ageing (with or without thermocycling) and the power of the curing unit (high or low) (n=5). After surface treatments, Tygon tubes (0.75 mm inner diameter and 0.5 mm height) were positioned on the ceramic plates. The resin cements (RelyX ARC and RelyX Unicem 2) were inserted into the tubes and photoactivated at different light intensities (High or Low power modes) by 40 s. The specimens were stored in 100% relative humidity at 37º C for 24 h. Half of the specimens were tested immediately and the remaining were submitted to 10,000 thermocycles before test. Data were checked for homoscedasticity and analyzed using three-way analysis of variance (ANOVA) and Tukey HSD test ($p<0.05$). *Results:* A decrease in bond strength values was observed when specimens were submitted to ageing (thermocycling). When analyzing the photoactivation intensity, lower bond strength values were found with high power mode to both resin cements, except for RelyX Unicem 2 after ageing. *Conclusion:* Thermocycling reduced bond strengths of specimens; the self-adhesive resin produced higher bond strengths to the lithium disilicate glass ceramic; and low power photoactivation mode increased bond strengths for non-aged specimens.

Key-words: Bond strength, Dental ceramic, Microshear, Photoactivation, Resin cement, Thermocycling.

INTRODUCTION

The adhesive dentistry has been continuously improved with the development of new restorative materials and the use of different techniques, resulting in simplified clinical procedures with increased longevity. Several mechanical tests are routinely applied to evaluate the mechanical properties of the dental materials. Assays verifying bonding quality and strength of adhesive systems-resin cements to dental substrates-restorative materials are the tests most commonly employed for dental materials.[1]

Conventional tensile and shearing tests have been applied for several years, although it has been shown that the results obtained with these methodologies do not represent the bonding strength of the materials with precision.[2,3] This fact occurs because the specimens used in these tests are more susceptible to failures incorporated at the adhesive interface or at the bonded substrates due to their increased dimensions.[4] Furthermore, the loading method and the geometry of the specimens used on these methodologies can induce undesired stresses at the bonding interfaces, promoting great discrepancies on the results.[1,5-7]

Attempting to overcome the limitations of the bonding tests, a previous study evaluated the interfacial bond strength from smaller bond testing areas (1.0 mm^2).[8] This allowed obtaining several specimens from a single tooth, measuring the bond strength in different regions, while reducing adhesive failures with the microtensile test.[8] As in the tensile/microtensile methods, reduced adhesive regions were also introduced for the shearing methods in order to incorporate less failures and variables during the test. The shearing test using specimens with adhesive interfaces smaller than 1.0 mm^2 was initially proposed, being nominated "microshearing".[9] Another microshear bond strength (μSBS) modality was also suggested for checking the bond strengths of dental materials, using very small cylinders of resin-based cements (ø 0.75x0.5 mm) bonded to different substrates (dentin, enamel, ceramics, etc.).[10-12] With this methodology, it was possible to prepare multiple bonding specimens in a single ceramic surface or even at a single tooth region, without requiring additional procedures that may cause test variability, such as the trimming of the adhesive region necessary in many microtensile approaches.[13,14] Furthermore, a non-destructible aspect of this test makes this methodology less expensive, mainly for studies with ceramic substrates.

Nowadays, conventional and self-adhesive resin cements with dual-polymerization are available. These materials differ in their chemical formulations and luting clinical procedures. Briefly, self-adhesive resin cements simplified the luting technique by eliminating the needs for substrate pretreatment.[15] This approach seems to be an interesting alternative to conventional adhesive procedures.[16] However, detailed information is limited regarding the degree of conversion under different polymerization conditions and its effects on the bond strength.[15,17]

Immediate bond strength tests are reliable to assess adhesive capability, whereas long-term clinical trials are ideal to assessing the durability of adhesive materials.[16,18] However, several factors as high cost, patient compliance and time, obstruct their extensive use.[18] Therefore, *in vitro* artificial ageing techniques have been proposed to accelerate the degradation of interfaces and, hence, enable the measurement of the long-term bonding and durability of dental materials.[16] Accelerated *in vitro* bonding degradation strategies can be performed because of the action of water storage, temperature changes, and mechanical and/or load cycling.[16,19] The hydrolytic degradation process during water storage occurs due to water sorption and the solubility of resin-based materials, thus reducing the lifetime of dental restorations.[16]

Thus, the aim of this study was to evaluate the effect of the thermocycling and photoactivation on the bond strength of two resin cements, conventional and auto-adhesive, to a lithium disilicate-based glass ceramic using microshear test. The hypotheses tested were that: (1) ageing (thermocycling) would reduce bond strength values; (2) decreased photoactivation power would decrease bond strengths; and (3) different resin cements would promote different bond strength values.

MATERIALS AND METHODS

A self-adhesive cement (RelyX Unicem 2, shade A2, 3M ESPE, St. Paul, MN, EUA) and a conventional cement (RelyX ARC, shade A2, 3M ESPE) were used. Forty lithium disilicate-based glass ceramic plates (IPS e.max Press, Ivoclar-Vivadent, Schaan, Liechtestein), with 8.0 x 8.0 x 2.0 mm (*height* x *width* x *thickness*), were produced and randomly divided according to the resin cement (conventional or self-adhesive), the ageing (with or without thermocycling) and the light intensity (high or low power modes) of the curing unit (BluePhase G2, Ivoclar-Vivadent) (n=5).

The lithium disilicate glass ceramic plates were etched with 10% hydrofluoric acid (Condicionador de Porcelana, Dentsply, Petrópolis, RJ, Brazil) for 20 s, followed by water-rinsing for 60 s, and air-drying for 30 s. Afterwards, a silane coupling agent (Ceramic Primer, 3M ESPE) was applied in two layers on the ceramic surface and left to react for 1 min, followed by the application of a thin layer of a 2-step etch-and-rinse adhesive system (Single Bond Plus, 3M ESPE) and light curing for 20 s. Micro bore tygon tube (TYG-030, Small Parts Inc., Miami Lakes, FL, USA) molds with 0.75 mm inner diameter and 0.5 mm height were positioned on the ceramic plates with 2.5 mm separating space between tubes, and prepared to adhesive procedures.[20] The resin cements were manipulated according to manufacturer's instructions and inserted into the molds followed by 40 s photoactivation according to the groups (high or low power) using the pre-programed modes of a LED curing unit (Bluephase G2, Ivoclar). The light intensity of the LED device was verified according to the power modes with digital radiometer (Hilux Light Meter, First Medica, Greenshore, NC, USA). The high power mode presented 1,100 mW/cm^2 light output, and the low power mode 700 mW/cm^2. After, the specimens were stored in 100% relative humidity at 37º C for 24 h. The tubes were carefully removed using a surgical blade.

Thus, half of the specimens from each group were immediately tested and the other half submitted to 10,000 thermocycles (MSCT-3, Marcelo Nucci ME Instrument, São Carlos, SP, Brazil) in water baths at 5 and 50º C with 30 s dwelling time. For performing the μSBS test, a metallic jig was coupled to a mechanical test machine (EZ-Test, Shimadzu, Tokyo, Japan) and the ceramic plate was placed on the device, so that the resin cylinders specimens were positioned perpendicular to 0.2 mm in diameter looped-orthodontic wire (NiCr, Morelli, Sorocaba, SP, Brazil). The wire was placed around one of the resin cylinders and the microshear testing was performed by stressing specimens to failure under tension at 0.5 mm/min. The fracture surface of the specimens were analyzed under scanning electron microscopy (LEO 435 VP; LEO Electron Microscopy Ltd., Cambridge, UK) to determine the failure modes, classified as adhesive, cohesive in cement, cohesive in ceramic or mixed.

Data were analyzed for homoscedasticity using Shapiro-Wilk test. Three-way analysis of variance (ANOVA) was applied to establish the significance of differences between testing groups followed by Tukey HSD test. All tests were performed at 95% confidence level using a statistical software package (Sygma Plot 12, Systat Software Inc., San Jose, CA, USA).

RESULTS

The three-way ANOVA showed significant differences for the testing time ($p<.001$), resin cement ($p=.034$) and interaction between photoactivation power and testing time ($p<.001$). Microshear bond strength results were significantly different for the resin cements (RelyX ARC- 30.44±11.63 MPa; and RelyX Unicem 2- 33.83±10.30 MPa; $p=.034$) and ageing (Immediate- 37.18±11.03 MPa; and Thermocycled- 27.94±9.38 MPa; $p<.001$).

Table 1 shows that the bond strength values for the RelyX ARC photoactivated in low power mode were significantly lower than in high power mode when thermocycling was performed. Relyx ARC photoactivated in low power mode resulted in higher bond strengths when test was performed immediately (non-thermocycled).

Table 1 – Mean bond strength values (MPa) and standard deviations (±) for RelyX ARC experimental groups.

		Testing time (Ageing)	
Resin cement	Power mode	Non-thermocycled	Thermocycled
RelyX ARC	Low	40.55±10.34Aa	20.71±7.60Bb
	High	32.08±10.55Ab	30.09±9.88Aa

* Different capital letters indicate significant difference between columns and different small letters indicate significant difference between rows (p<.05).

Table 2 shows that the bond strength results for the RelyX Unicem 2 presented similar behavior to RelyX ARC.

Table 2 – Mean bond strength values (MPa) and standard deviations (±) for RelyX Unicem 2 experimental groups.

		Testing time (Ageing)	
Resin cement	Power mode	Non-thermocycled	Thermocycled
RelyX Unicem 2	Low	41.44±11.81Aa	26.00±9.23Bb

	High	35.36±9.20Ab	33.62±5.64Aa

* Different capital letters indicate significant difference between rows and different small letters indicate significant difference between columns ($p<.05$).

Mixed failure mode was the most predominant, followed by adhesive mode. Very low incidence of cohesive failures within cement occurred (Figure. 1).

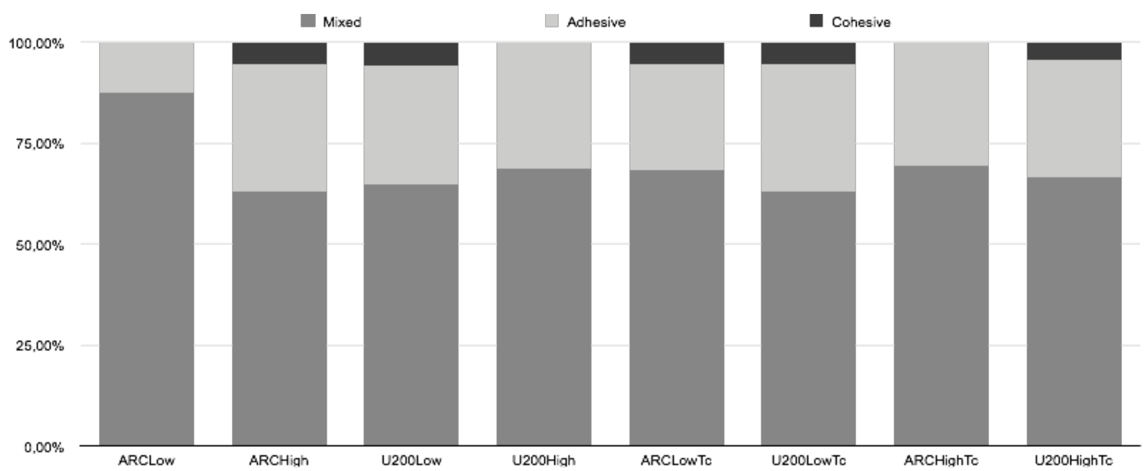

Figure 1 – Plot of failure modes for each group showing resin cement cohesive failures, adhesive failures and mixed failures.

DISCUSSION

The hypothesis proposed that the thermocycling would reduce the bond strength values was rejected since the statistical analysis revealed significant differences test time ($p<.001$), but samples activated with higher potency were not affected by ageing (Tables 1 and 2). The power mode of phtoactivation was not significant to affect the bond strength, when the thermocycles were performed the bond strength decrease to the lower power mode (Tables 1 and 2) rejecting the hypothesis that decreasing photoactivation power would lower bond strengths. Finally, the hypothesis that different resin cements would result in different bond strength values was accepted.

Aging methods such as long-time water storage, thermo-cycling, and pH cycling have been widely used to evaluate the stability of resin-dentin bonds.[21,22] Thermocycling showed a significant effect on the μSBS values, indicating that this ageing method was effective to accelerate the degradation of the adhesive interface. One important reason for using thermocycling instead of water storage alone is the testing time. Previous studies showed that even the flexural strength of ceramic materials might decrease under thermocycling regimes, commonly encountered in the oral environment, due to the extension of larger flaws on the ceramic surface.[22,23] Alternating hot and cold temperatures can produce detrimental tensile stress on the ceramic surface, resulting in crack growth, thus leading to a decrease in the mechanical strength.[22,23]

In this study, differences were found between the groups according to the ageing performed, since the thermocycling reduced the microshear bond strength values. In contrast, the investigation of Xiaoping *et al.* (2014) revealed no significant changes between testing groups after thermocycling probably because alternating temperature cycles did not result in growing of surface crack, probably because of the 3-dimensional interlocking structure of the lithium disilicate crystals, which withstood temperature fatigue caused by 10,000 times. This microstructure prevented the forming of new cracks and propagating of original cracks on the ceramic surface.[24] Additionally, the polymerization shrinkage of resin cements may prevent surface flaws from being extended following thermocycling by imposing a compressive stress on the ceramic surface.[24,25] Other possibility would be that the luting of resin cement on the ceramic surface could heal the surface cracks and form a barrier which could prevent water stress corrosion to ceramic effectively and ensure a longer working life of all-ceramic restorations in the oral environment.[24]

Reducing the photoactivation power intensity, the bond strength significantly increased, except for the RelyX Unicem 2 specimens after thermocycling. This fact may be explained by the freedom of monomers assured by the lower energy emitted when the low power mode was used. This probably results in better conversion of monomers in polymers and increased bond strength promoted by the association of the slow polymerization reaction with the improved activity of the chemical phase of the dual-cure resin cements.[26] Additionally, possibly the reduced speed of the polymerization reaction can also lead to lower shrinkage stress, thus resulting in better bond strengths due to a more intact adhesive interface.

The self-adhesive resin cement showed higher bond strength values than the conventional resin cement (RelyX ARC- 30.44±11.63 MPa; and RelyX Unicem 2- 33.83±10.30 MPa; p=.034). These results may be explained by improved chemical bonds of the MDP phosphate monomers to the ceramic oxides.[27] Moreover, a possible chemical incompatibility between adhesive systems with low pH and chemically and dual-cured resin materials was related.[27] It is known that acidic resin monomers retard the polymerization of chemically/dual-cured composites that are initiated via peroxide-amine type binary redox catalysts, this fact would explain the lower bond strengths verified for the conventional dual-cured resin cement, which is dependent of surface treatments with adhesive systems previous to luting procedures. [27]

The research design of this study presents some intrinsic limitations, such as the *in vitro* analysis only. Future studies with microtensile bond strength test, which overcome these limitations, will be of benefit.

CONCLUSIONS

The present study showed that:

(1) ageing of the specimens by thermocycling affect the bond strength of both conventional and self-adhesive resin cements to lithium disilicate glass ceramic;

(2) the self-adhesive resin cement was more effective to bonding to the glass ceramic;

(3) and low photoactivation power intensity increased the bond strength of resin cements.

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CONCLUSÃO

A verificação da resistência de união de materiais restauradores a diferentes substratos também figura como ensaio mecânico de grande importância, sendo que o teste de microcisalhamento tem sido bastante utilizado com este propósito em diversos estudos (43-46). Entretanto, assim como alterações nas configurações do teste de microtração resultam em valores discrepantes de resistência de união(27, 37), modificações realizadas no teste de microcisalhamento também podem gerar dados divergentes.

Os resultados encontrados no presente estudo mostram que diferentes métodos de ensaio para obtenção da resistência de união entre cerâmicas odontológicas e cimentos resinosos podem ser utilizados. O estudo com análise por elementos finitos (Capítulo 2) mostrou uma maior fidelidade ao que o ensaio se propõe para os testes de microtração, já que os mesmo apresentaram altas tensões de tração na interface, enquanto o ensaio de microcisalhamento apresentou tensões perpendiculares ao sentido do ensaio. A investigação in vitro sobre os teste de resistência de união (Capítulo 2), revelaram maiores valores de resistência de união para os espécimes testados por microcisalhamento, o que pode indicar menor incorporação de falhas na região adesiva. Outros aspectos estudados, como diferentes cimentos e potencia de fotoativação não provocaram diferenças estatísticas.

Embora no Capítulo 1, onde o ensaio de microcisalhamento foi utilizado para avaliar a resistência de união em diferentes aspectos, a diminuição da potencia de fotoativação desempenhou um fator de aumento na resistência de união, o que pode ser definido como uma maior liberdade dada aos monômeros de se movimentarem na matriz do cimento e como conseqüência, possivelmente um maior grau de conversão dos monômeros em polímeros. Outro fator estudado, os cimentos resinosos, também apresentaram diferenças entre os grupos testados, isso se deu devido à presença de monômeros fosfatados que auxiliam na união dos cimentos a óxidos cerâmicos. E, finalmente, o envelhecimento das amostras por meio de termociclagem, que, de acordo com a literatura, promove tensão na interface o que diminui a resistência de união entre os materiais, assim como ocorreu nesta investigação.

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